OIL RECOVERY FROM PALM OIL SOLID WASTES

DASHINY D/O GOPAL

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I declare that this thesis entitled “Oil Recovery from Palm Oil Decanter Cake and Spent Bleaching Clay.” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : ……..

Name : ……..

Date : ……..

Special Dedication of This Grateful Feeling to My Beloved father and mother;

Mr. GOPAL S/O BALAKRISHNAN and Mrs. LALITHA D/O RAMASAMY

Loving siblings;

G. KUMARESWARAN AND G.SAMIHETA

Supportive families;

Uncles and Aunties

For Their Love, Support and Best Wishes.

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ABSTRAK

Kajian selama beberapa tahun telah dilakukan ke atas pengekstrakkan sisa minyak dari bahan buangan pepejal minyak kelapa sawit. Kek decanter ialah bahan buangan pepejal yang dihasilkan daripada kilang pengisar minyak kelapa sawit selepas air buangan industry minyak kelapa sawit dimampatkan, manakala tanah peluntur yang telah digunakan ialah bahan buangan pepejal hasil daripada penapisan minyak kelapa sawit. Biasanya, bahan buangan ini masih mengandungi 30-40% minyak dan bahan buangan pepejal sekarang ini dibuang terus di tempat buangan sampah tanpa dirawat di mana kerana perbuatan ini, pencemaran air dan udara yang serius berlaku. Pemulihan minyak dan penggunaan semula tanah liat peluntur yang telah digunakan serta kek decanter membuka peluang yang besar untuk menjimatkan kos dalam industry memproses minyak ini. Kajian ini menjelaskan tentang pengekstrakkan sisa minyak menggunakan tanah peluntur yang telah digunakan daripada penapisan minyak kelapa sawit. Pengekstrakkan sisa minyak dari kilang pengisar minyak kelapa sawit juga dijelaskan dalm kajian ini. Di sini, dua kaedah digunakan membandingkan pengekstrakkan minyak daripada kek decanter dan juga tanah liat yeng telah digunakan. Satu ialah kaedah pengekstrakkan soxlet dan satu lagi ialah pengekstrakkan pelarut kaedah lama. Perbandingan dua kaedah menunjukkan pengekstrakkan soxlet boleh member hasil yang banyak. Suhu optimum dan masa terbaik untuk mengeringkan kek decanter dan tanah liat yang telah digunakan juga ditentukan. Untuk kes ini, sampel-sampel dikeringkan dengan suhu 60^0C- 110^0C, dan suhu yang terbaik ialah 90^0C. Masa optimum telah ditentukan selama 12 jam. 4 pelarut yang berbeza telah digunakan untuk mengekstrak sisa minyak daripada sampel bahan buangan. Keputusan menunjukkan bahawa peratus pengekstrakkan minyak daripada metil etil ketone dan acetone lebih tinggi berbanding dengan hexane dan petroleum ether. Nilai iodin juga ditentukan untuk membandingkan kualiti miyak yang diekstrak. Nilai iodin yang diperoleh adalah dalam lingkungan 40-80. Mengikut analisis PORIM, nilai min minyak mentah ialah 51.3.

ABSTRACT

Many years of research have been done on extraction of residue oil from palm oil solid wastes. Decanter cake is the solid waste produced from palm oil milling company after decanting the palm oil mill effluent, while spent bleaching clay is the solid waste from palm oil refinery. Basically, this wastes still contains 30-40% of oil and this solid wastes are currently disposed directly in landfills without treatment, causing severe water and air pollution problems. Recovery of oil and the reuse of spent bleaching clay and decanter cake is the areas where great opportunity exists for cost saving in the oil processing industry. This study described the extraction of residual oils of spent bleaching earth (SBE) from palm oil refinery and also described the extraction of residue oil from palm oil milling industry. Here, two methods are used for comparison to extract the oil from decanter cake and also spent bleaching clay. There are soxhlet extraction method and also solvent decanting method. The comparison of two methods shows that soxhlet extraction can give higher yield. The optimum temperature and best duration of time to dry the decanter cake and spent bleaching clay also determined. In this case, the samples were dried at the temperatures 60^0C- 110^0C, and the best temperature is 90^0C. The optimum time was determined as 12 hours. Four different solvents were used to extract the residue oil from the waste samples. The results shows that the percentage of oil extracted from Methyl ethyl ketone and acetone was high compared to hexane and petroleum ether. The iodine value was also determined to compare the quality of the oil extracted. The range of iodine value obtained was 40-80. According to PORIM analysis mean value of crude oil is 51.3.

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LIST OF SYMBOLS

M	-	Molarity
m	-	Mass
N	-	Normality
V	-	Volume

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CHAPTER 1

INTRODUCTION

1.1 Overview of Research

The oil palm tree (Elais guineensis) originated from West Africa andwas introduced in Malaysia in early of 1870's. Today Malaysia is the largest producer and exporter of palm oil in the world. According to Malaysian Palm Oil Council currently we are producing 51% of world palm crude edible oil and accounts for 62% of world exports. Oil palm covers 67% of Malaysia's total agricultural land, and 500,000 people in Malaysia are engaged in the sector.

Palm oil is produced from palm fruit in mills through steaming and squeezing process. Figure 1.1 shows the palm oil tree, its fruit and also the cross sectional of the palm oil fruit. Each tree produces compact bunches weighing between 10 to 25 kilograms with 1000 to 3000 fruits per bunch. An individual palm fruit consists of a hard kernel (seed) inside a shell (endocarp) which is surrounded by thin fleshy mesocarp. Mesocarp is the part where we can extract crude palm oil.

Figure 1.1 Palm Oil fresh fruit bunch

The oil palm produces two types of oils; crude palm oil (CPO) from the fibrous mesocarp and crude palm kernel oil from the kernels. Besides that, the industry also produces large amounts of by-products such as palm kernel cake, palm oil sludge (decanter cake), empty fruit bunches and palm pressed fiber as residues.

The problem now in Malaysia is to manage the wastes generated during the palm fruit processes. From the figure 1.2 as presented below, we can see that on average one tone of crude palm oil is extracted from 5.8 ton of fresh fruit bunch. Fibre, shell, decanter cake and empty fruit bunch are 1.42 ton, 0.35 ton, 0.18 ton and 1.63 ton respectively. Empty fruit bunch is the bulk of solid residue and it isuse as a fuel for boiler and also used as a substrate for mushroom and as organic fertilizer. Palm fibres are mainly used as fuel for boilers (heating value of <5 MJ/kg dry fibres). Palm shells are used as feedstock of activated carbon due to undesirable emissions. Decanter cake is used as a fertilizer. Lastly, palm oil mill effluent which is the mixture of polluted effluent, is treated and digested anaerobically to yield biogas, which is used in modified diesel engine with a 90 kW induction motor. (Somporn et. al, 2004)

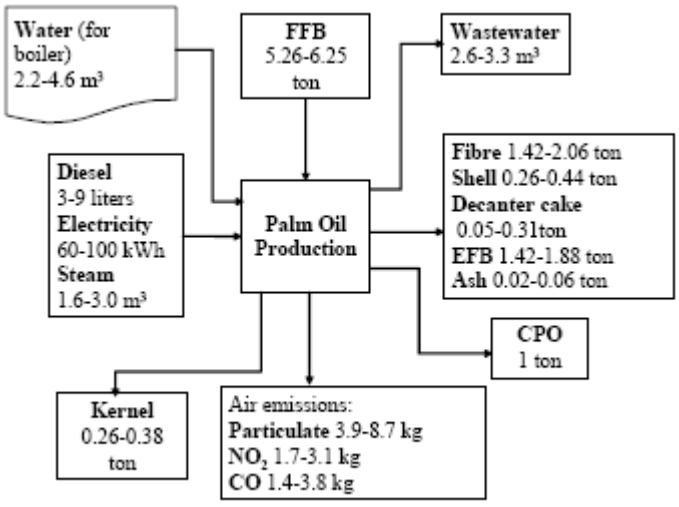

Figure 1.2 Unit process of palm oil production.

Palm oil sludge or decanter cake is the material that remains after decanting the palm oil effluent. Decanter cake produced after the oil mesh from digester passes through the centrifugal decanter to purify the oil before sending to a storage tank.

Decanter cake can be filter pressed before dried to produce dehydrated palm oil mill effluent. It can be centrifuged in the wet state after undergone anaerobic, thermophilic and acidophilic fermentation to produce fresh centrifuged sludge solid. Currently this sludge solid is used as fertilizers since it contains amino acid, crude protein and fibre.

The loss of oils in palm oil decanter cake is a concerned by the palm oil industry, as the oil can be used for industrial purposes. The residue oil in decanter cake should be recovered and re-used as raw material for industrial application. This somehow will save cost in the oil industry. One best approach is to convert the oil into fatty acid alkyl esters. Here , we need to treat decanter cake in organic (alcohol) phase at high temperature (110-270^{0}C) under moderate pressure to recover the oil and convert it into alkyl esters of C1 to C8 carbon atoms. The oil will be converted insitu to alkyl esters using methanol. The alkyl esters are very useful ingredients in non-food applications. (Loh et. al,2006)

1.2 Problem Statement

The decanter cake contains high percentage of oil. Disposal of decanter cake by incineration, inclusion in animal feeds, land filling method or concrete manufacturing is currently practiced by most of the palm oil milling companies. But, large quantity of decanter cake is disposed off in landfills, only causing fire and pollution hazards due to the residue oil content in the earth. So, the oil in the decanter cake would be just wasted if the oil was not recovered and this situation will cause loss of oil in the future.

In the refining of palm oil, bleaching clay dosages of 0.5-1% are generally used. From, Malaysia alone, with a production of 9 million tons of oil in 1999, a total of 70,000 tons of spent bleaching clay is estimated to be generated yearly. Spent bleaching clay contains about 30-40% of oil by weight of spent bleaching clay and this constitutes a major loss in oil as well a major cost from the clay since the

spent clay is currently disposed untreated. In addition, the use and disposal of the spent bleaching clay is becoming a potential problem in the producing countries because of the rapid growth of the industry and currently, oil-laden spent bleaching clays are mainly disposed of in landfills or in waste dumps, as the spent clays are considered nontoxic.

Extracting oil from palm oil decanter cake has never been reported before. Now decanter cake is only used as fertilizers without extracting the residue oil. So, here we can extract the oil and can be used for so many purposes. As a suggestion the crude oil extracted can be converted to biodiesel because the oil extracted from palm oil solid waste has poorer quality compared to crude palm oil. After extracting oil, residue solid waste can be used as fertilizer or animal feed.

Utilization of palm oil decanter cake also will improve the environment. This is because the disposal of sludge solid waste will increase the Biochemical Oxygen Demand (BOD) of the land. Other than that, land filling the sludge solid is expensive.

The highly increasing trend of oil price in this year has had a great impact on both agricultural and industrial sectors. This is because, increasing oil price also increasing cost of production. So in this situation Malaysian government needs to do researches on new sources of energy to substitute petroleum products. Using biodiesel is the best way to reduce foreign import of petroleum products. Furthermore there are a lot of advantages using biodiesel instead of conventional diesel fuel which are environmental benefits, less toxicity for humans because the raw materials used to produce it are natural and renewable making it biodegradable and non-toxic.

1.3 Objective of Study

The objectives of the studies are as follows;

- To determine the optimum temperature and optimum drying time to remove higher percentage of moisture from palm oil solid wastes.
- To extract crude oil from palm oil decanter cake by solvent decanting extraction and soxhlet extraction.
- To determine the higher percentage of oil yield using methyl ethyl ketone, acetone, hexane and petroleum ether as solvents.
- To determine the iodine value and acid value of the oil extracted from palm oil decanter cake and spent bleaching clay.

1.4 Scopes of study

Based on the objectives, the main scope of this project is an overview of the utilization of palm oil sludge solid in industry. The scope of study will involve collecting oil palm decanter cake sample from a nearby palm oil milling company and spent bleaching clay from palm oil refining company. Then, drying the sample with different temperature to remove moisture and preparation of the sample for extraction. This temperature parameter is to determine the optimum temperature to dry decanter cake to extract maximum oil. Then the solvent extraction method will be carried on using different type of solvents, metylethylketone, acetone, petroleum ether and hexane. There will also be a comparison between solvent decanting extraction method and soxhlet extraction method .This is to determine the effect of the solvent on the % of extracted oil. The effect of the solvent, extraction time, solvent/feed ratio, and mixing rate on the extraction efficiency was investigated through this work.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction and Historical Background

Chemical analyses play an important role in our life, from agricultural, environmental, clinical, pharmaceutical chemistry, oleo chemistry, toxicology, manufacturing, metallurgical, archaeology, to forensic. The value of a piece of land is determined by the content of carbon and nitrogen. The quality of air is determined by analyzing the percentage of the pollutants composition. The price of the coal will be determined by the percent of sulfur impurity present.

We look into our material world through the methods and the tools of analytical chemistry. Those analytical techniques existing now are always being sought to improve by analytical chemists to meet the arising requirements for better chemical measurement from our society. Usually one or more standard specific procedures are available for determination of an analyte in a provided sample. However, it is not exactly can be accepted by the other analyte. The analyst needs to depend on his experience and knowledge to carry out and analytical method for a sample. Since oil palm dominate most of the needs in our life, getting to know the process involve in oil extraction is important. The quality of oil is determined by the composition of saturated and unsaturated fatty-acids. Palm oil has higher saturated fatty acids and this make the oil more stable and less prone to oxidation at higher temperature.

2.2 The oil palm

Elaeis Guineensis Jacq is the scientific name of oil palm and this is the most important species in the genus Elaeis which belong to the family Palmae. Oil palm (*Elaeis guineensis*) is grown extensively in Southeast Asia and Equatorial Africa and it produces more oil per area than any other plant. (Poku et.al, 2002.) Oil palms start bearing bunches 2 ½ - 3 years after field planting. The usual frequency of a harvesting round is 10 to 15 days or 2-3 times a month. The fruit bunches are generally transported to palm oil mill on the day of harvesting.

The palm oil mil production capacity is around 45 tons of FFB per hour or around 1000 tons of FFB per day. The mill operates approximately 16-24 hours per day. (Mahlia et.al, 2000). Below are the technological process involve in extracting oil from the palm oil fresh fruit bunches and Figure 2.1 explains the palm oil milling process in a process flow diagram.

Loading ramp : After passing over the weighbridge the fruit has to be held for a time until it can enter the first stage of processing. For loading, ramp is the place where the FFB are transported and unloaded in the mill.

Sterilization : This process is done batchwise in an autoclave for 1 hour 40 minutes for the Fresh Fruit Bunch to be completely cooked. The temperature inside is about 120-130^{o}C. The steam condensate is the waste water generated at this step.

Stripping (threshing) : This process is to separate the sterilized fruits from bunch stalks. This process generates the empty fruit bunches (EFB).

Digestion : The separated fresh fruits are put into the place where they mashed under steam heated conditions. No residues occurred on this step.

Crude palm oil extraction: the homogenous oil mash from the digester is pushed through a screw press, and later passes through a vibrating screen, a hydrocyclone and decanters to remove fine solids and water. Decanter wastewater and decanter cake are the major wastes at this step. Centrifugal and vacuum driers are used to further purify the oil before sending it to a storage tank. The temperature of oil (60°C) in the storage is maintained with steam coil heating before the CPO is sold.

Nut/ Fibre Separation: the fibre and nuts from the screw press are separated in a cyclone. The fibre that passes out of the bottom of the cyclone is used as boiler fuel from which ash is produced after combustion.

Nut Cracking: the nuts are cracked in a centrifugal cracker. After the cracking process, the kernels and shells are separated by clay suspension (Kaolin). The separated shells from the kernels are sold to other mills as fuel. The kernels are sent to the kernel drying process in a silo dryer to sell (for extraction) to other mill.

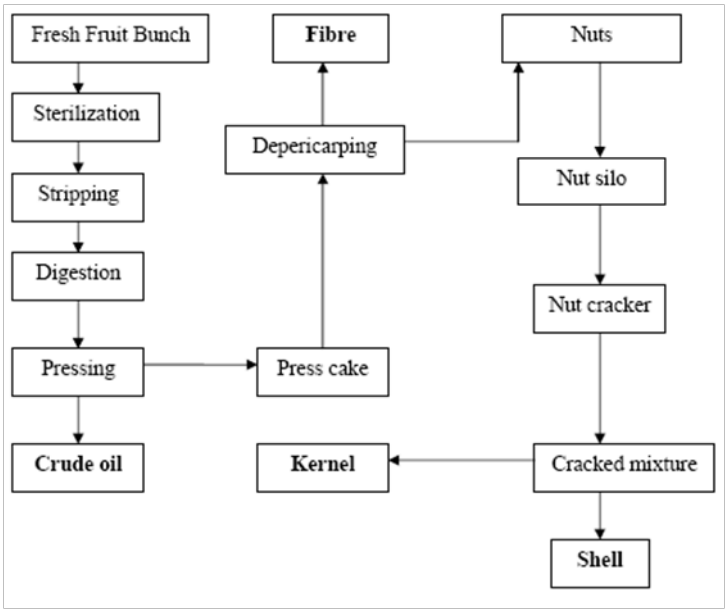

Figure 2.1 Process flow diagram of palm oil extraction

2.3 Characteristics of palm oil

The oil palm produces two types of oils, palm oil from the fibrous mesocarp and lauric oil from the palm kernel. In the conventional milling process, the fresh fruit bunches are sterilized and stripped of the fruitless which are then digested and pressed to extract the crude palm oil (CPO). The nuts are separated from fiber in the press cake and cracked to obtain palm kernels which are crushed in another plant to obtain crude palm kernel oil (CPKO) and a by-product, palm kernel cake which is used as an animal feed. Obtained palm kernels which are crushed in another plant to obtain crude palm kernel oil (CPKO) and a by-product, palm kernel cake which is used as an animal feed.

2.3.1 Chemical properties of palm oil

Crude palm oil is one of the major sources of vitamin E and contains high quantities of tocopherols and tocotrienols in the range of 600-1000 ppm. (Marsin et. al, 2005). Palm oil has a balanced ratio of saturated and unsaturated fatty acids while palm kernel oil has mainly saturated fatty acid which is broadly similar to the composition of coconut oil.

Palm oil is semi-solid at room temperature. In its virgin form, the oil is bright orange-red in color due to its high content of carotene. Triglycerides forms the major component and bulk of the glyceridic material present in palm oil with small amounts of monoglycerides and diglcerides, which are artifacts of the extraction process. Knowledge about the detailed structures of the triglycerides present in palm oil is important, because they define the physical characteristics of the oil. The melting points of triglycerides are dependent on the structures and the position of the components acids present. They also effect the crystallization behavior of the oil. The semisolid nature of the palm oil at room temperature has been attributed to the presence of the oleo-disaturated fraction.

Palm oil contains palmitic acid (a fatty acid made by our body), the monounsaturated oleic acid, polyunsaturated linoleic acid (an essential fatty acid) and stearic acid. The typical blend in palm oil is 45% palmitic, 40% oleic, 10% linoleic and 5% stearic. Palm oil (and its products) has good resistance to oxidation. The palm oil is also rich in natural chemical compounds important for health and nutrition. Among others, it is a natural source of carotenoids and vitamin E as well as supplying fatty acids and other important fat-soluble micronutrients. The carotenoids, tocopherols, sterols, phosphatides, triterpenic, and aliphatic alcohols form the minor constituents of palm oil, and play significant role in the stability and refinability of the oil, in addition to increasing the nutritive value of the oil.

Crude palm oil contains between 500-700 ppm of carotenoids mainly in the forms of α- and β- carotenes. In crude palm oil , the presence of these carotenoids appears to offer some oxidative protection to the oil through a mechanism where they are oxidized to the triglcerides. The combined effects of the properties of carotenoids, tocopherols, tocotrienols and the 50% unsaturation of the acids confer on palm oil a higher oxidative stability compared to other vegetable oils. (Fereidoon,2004). Compared to soy oil, palm oil has a higher amount of saturated fatty acids but this makes it more stable and less prone to oxidation at high temperatures. In terms of sterols, palm oil contains far less cholesterol than other vegetable oil as shown in Table 2.1 below. The lower cholesterol levels in crude palm oil add nutritive value and advisable to be used in food industries.

Table 2.1: Cholesterol levels in crude oils and fats. Source: (Fereidoon,2004)

Oil type	**Average (ppm)**	**Range (ppm)**
Coconut oil	14	5-24
Cocoa butter	59	n.a
Palm kernel oil	17	9-40
Palm oil	18	13-19
Sunflower oil	17	8-44
Soybeen oil	28	20-35